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Innovative Destruction of Complex Industrial Wastes - Auto Oxidation of Tannery Beamhouse Wastewater

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16. ABSTRACT

This report documents a study of the effectiveness of an auto-oxidation technique for treating potentially toxic pollutants from a manufacturing source. The particular waste used for evaluation was a tannery unhairing effluent.

Tannery unhairing waste was treated in a pilot plant scale auto-oxidation unit designed and built by Technical Associates for Industry, Inc., Red Bank, New Jersey 07701. This waste is highly alkaline with a pH generally higher than 11 and has a COD greater than 50,000 mg/liter, consisting largely of protein and sulfide. The auto-oxidation pilot plant in static tests effectively and rapidly removed sulfide from the waste. Removal of COD, nitrogen, and suspended solids was considerably less effective. Combining the auto-oxidation with ultraviolet light and a hydrogen peroxide addition improved the COD removals very little.

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by

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FOREWORD

When energy and material resources are extracted, processed, converted, and used, the related pollutional impacts on our environment and even on our health often require that new and increasingly more efficient pollution control methods be used. The Industrial Environmental Research Laboratory - Cincinnati (IERL-Ci) assists in developing and demonstrating new and improved methodologies that will meet these needs both efficiently and economically.

This report documents an evaluation of auto-oxidation treatment of a difficult leather processing waste stream. The study was undertaken to determine whether auto-oxidation would be effective in treating a complex industrial wastewater. The results provide information useful to industry in the development of economic wastewater treatment. For further information on the subject, contact the Industrial Pollution Control Division of the Industrial Environmental Research Laboratory, Cincinnati, Ohio 45268.

David G. Stephan
Director
Industrial Environmental Research Laboratory
Cincinnati

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ABSTRACT

This project was intended to obtain data on the effectiveness of a novel destruction technique for treating potentially toxic pollutants from a wide range of manufacturing sources. The particular waste used for evaluation was a tannery unhairing effluent. The novel technique examined was an auto-oxidation process.

Tannery unhairing waste was treated in a pilot plant scale auto-oxidation unit designed and built by Technical Associates for Industry, Inc., Red Bank, New Jersey 07701. This waste is highly alkaline with a pH generally higher than 11 and has a COD greater than 50,000 mg/liter, consisting largely of protein and sulfide. The auto-oxidation pilot plant in static tests effectively and rapidly removed sulfide from the waste. Removal of COD, nitrogen, and suspended solids was considerably less effective. Combining the auto-oxidation with ultraviolet light and a hydrogen peroxide addition improved the COD removals very little.

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(4-76)

CONTENTS

Disclaimer ii
Foreword iii
Abstract iv
Figures vi
Acknowledgments viii

1. Introduction 1
2. Conclusions 3
3. Materials and Methods 4
4. Experimental Procedures 6

References 27

FIGURES

Number		Page
1	Schematic of auto-oxidation pilot plant	5
2	Results of static test in pilot plant. 15:1 dilution of unhairing effluent	7
3	Results of static test in pilot plant. 20:1 dilution of unhairing effluent	8
4	Reduction of sulfide concentration in static test of pilot plant on sodium sulfide/lime solution	9
5	Results of continuous feed test in pilot plant. 10:1 dilution of unhairing effluent	11
6	Results of continuous feed test in pilot plant including analysis of feed solution. 10:1 dilution of unhairing effluent.	12
7	Results of continuous feed of settled unhairing effluent diluted 10:1	13
8	Bench test of additives to tannery waste. Control.	14
9	Bench test of additives to tannery waste. Effect of ultraviolet light	15
10	Bench test of additives to tannery waste. Effect of hydrogen peroxide	16
11	Bench test of additives to tannery waste. Effect of ultraviolet light and hydrogen peroxide	17
12	Bench test of additives to tannery waste. Effect of copper catalyst	18
13	Bench test of additives to tannery waste. Effect of ultraviolet light and copper catalyst	19
14	Bench test of additives to tannery waste. Effect of hydrogen peroxide and copper catalyst	20

15 Bench test of additives to tannery waste. Effect of hydrogen peroxide, ultraviolet light, and copper catalyst. 21

16 Effect of addition of hydrogen peroxide to a continuous feed pilot plant test. 10:1 dilution of unhairing effluent. 23

17 Effect of addition of hydrogen peroxide to a continuous feed pilot plant test. 20:1 dilution of unhairing effluent. 24

18 Effect of addition of ultraviolet light followed by hydrogen peroxide to a continuous feed pilot plant test. 10:1 dilution of unhairing effluent 25

19 Effect of addition of hydrogen peroxide followed by ultraviolet light to a continuous feed pilot plant test. 10:1 dilution of unhairing effluent 26

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In addition, I express appreciation to Dr. I. Chang of Cheney State College and many of my coworkers who assisted in both discussions and analyses of samples including J. E. Cooper, J. Cheskiewicz, and M. Tunick.

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SECTION 1

INTRODUCTION

In 1968 the Hides and Leather Laboratory was approached by Technical Associates for Industry (TAFI) with a proposal to test an innovative process for treatment of tannery lime-sulfide unhairing wastes. TAFI is a small, private consulting firm. Their general interest was in treatment of industrial and municipal wastes to meet regulatory limits. Their experience to date, on treatment of wastes with this process, was on municipal sludges and industrial wastes with high carbohydrate content. The process is based on a free-radical, auto-oxidation mechanism of breakdown of the waste. The free radicals are produced by passing the solution under pressure through an orifice. The basis for free radical production proposed by N. Zaleiko, President of TAFI and inventor of the process, is cavitation of the solution during treatment. The collapse of gas bubbles in cavitation can develop very high local temperatures. Under adiabatic conditions, gas temperatures at the minimum bubble radius can reach 2,000°F (1). These local high temperatures can be expected to lead to ionization effects and the production of hydroxy-free radicals and peroxides. Extensive cavitation effects on the oxidation of phenol are described by Chen et al. (2).

This process has been developed over the past three years. The results have not been published, although free radical oxidation of municipal sludge by other methods has been proposed (3). The process is simple in design and is capable of further improvement in the oxidative degradation process by substitution of additional oxidants such as peroxide into the system. These oxidants are effective in destroying refractory organics. It is also possible to incorporate ultraviolet light into the system without changing the design. The original intent of this project was to carry out a side-by-side pilot scale comparison of the auto-oxidation process with an activated sludge process which this laboratory was then developing for the tanning industry. However, due to unexpected characteristics of the tannery waste and the short duration of this project, this report is limited to a description of the apparatus and the results of a number of trials performed.

The effluent stream from the unhairing process in a tannery is a difficult waste to treat by conventional methods. Typical composition of unhairing waste is: BOD, 20,000 ppm; COD, 40,000 to 60,000 ppm; solids, 50,000 ppm; pH, 12.5; and sulfide, 1,000 to 3,500 ppm (4). The beamhouse process (5) starts by soaking hides in water in order to rehydrate them and to remove salt. The soaked hides are then placed in a 2-4% calcium hydroxide suspension along with approximately 2% by weight of the hide of sodium sulfide in order to dissolve the hair. The hides may then be relimed with additional calcium hydroxide or simply washed. From this stage they continue into the actual tanning process. The effluent from the tanning process

is not included in the study. There was no a priori reason why this auto-oxidation process should be able to treat this particularly difficult waste. However, its investigation for this purpose was deemed to be worthwhile, because if it were successful, the benefits to the industry would be great. The tanning industry will be required to invest millions of dollars over the next five to ten years to meet effluent requirements (6). It was a significant increase in difficulty to go from treating municipal sludge to testing unhairing effluent. However, the proposal was made at a time when waste treatment research on tannery effluent was being conducted at the Department of Agriculture's Research Center in Philadelphia and raw waste, the necessary equipment for the needed analyses, and personnel were available and the decision was made to proceed with the proposal.

This will serve as the final report to EPA on this cooperative agreement.

SECTION 2

CONCLUSIONS

The innovative treatment of tannery wastes, using the oxidative pilot plant manufactured by TAFI, rapidly removed sulfide from tannery lime-sulfide unhairing effluent. Removal of COD, suspended solids, and nitrogen under the conditions studied was not effective. The excessive formation of foam in this waste might have been responsible. There were indications that additional research on a combination of this oxidative process, metallic catalysis, ultraviolet light, and hydrogen peroxide would prove to be more successful. This cooperative agreement did not provide sufficient resources to continue these experiments beyond what is presented here. No estimate of cost effectiveness was made to determine whether this process could be used commercially to treat beamhouse effluent.

SECTION 3

MATERIALS AND METHODS

The apparatus which is the basis of this innovative process, the oxidation pilot plant, is detailed in Figure 1. The material to be treated was pumped from a feed tank (250 gal) into a reservoir which was maintained at a level of approximately 18 gal. The treated solution flows out of the system through the overflow at the same rate that feed was pumped into the system. Solution from the reservoir was pumped by a 5 hp circulation pump through the oxidation nozzle and back to the reservoir through a 3/4" ID copper pipe at a rate of 5 gal/min. The oxidation nozzle was essentially a constriction in the recycle loop. As the solution flowed out of the nozzle, the pressure on the solution was rapidly reduced causing cavitation. At the same time air was incorporated into the loop to prevent the level of dissolved oxygen from dropping to zero. Two types of tests were run on the apparatus. The first, referred to as a static test, consisted of recycling the solution to be treated from the reservoir, through the pump and around the loop, through the oxidation nozzle and back into the reservoir. No additional feed was added. In a continuous run, the second type of run, after the system was filled, fresh solution to be treated was pumped into the reservoir and the overflow from the treatment was allowed to flow out into a final tank or into a drain. At a later stage of the research an ultraviolet lamp was added to the system positioned directly at the end of the oxidation nozzle. When used in the experiments, hydrogen peroxide was pumped into the system as indicated just before the pump. Metal salts, when added for catalysis, were added in the reservoir or dissolved in the feed tank. In a continuous run, the feed was typically added at a rate of 0.3-0.5 gal/hr. Samples were taken for evaluation of treatment from the overflow. The analyses were performed according to standard methods except as indicated. Analyses reported are:

1. Sulfide (s =),
2. Total Kjeldahl nitrogen using a Technicon autoanalyzer (n),
3. TOC (total organic carbon) using a Beckman TOC instrument,
4. COD (chemical oxygen demand) performed by the techniques developed by Oceanography and accepted by Standard Methods,
5. Suspended solids (ss), and
6. Volatile suspended solids (vs).

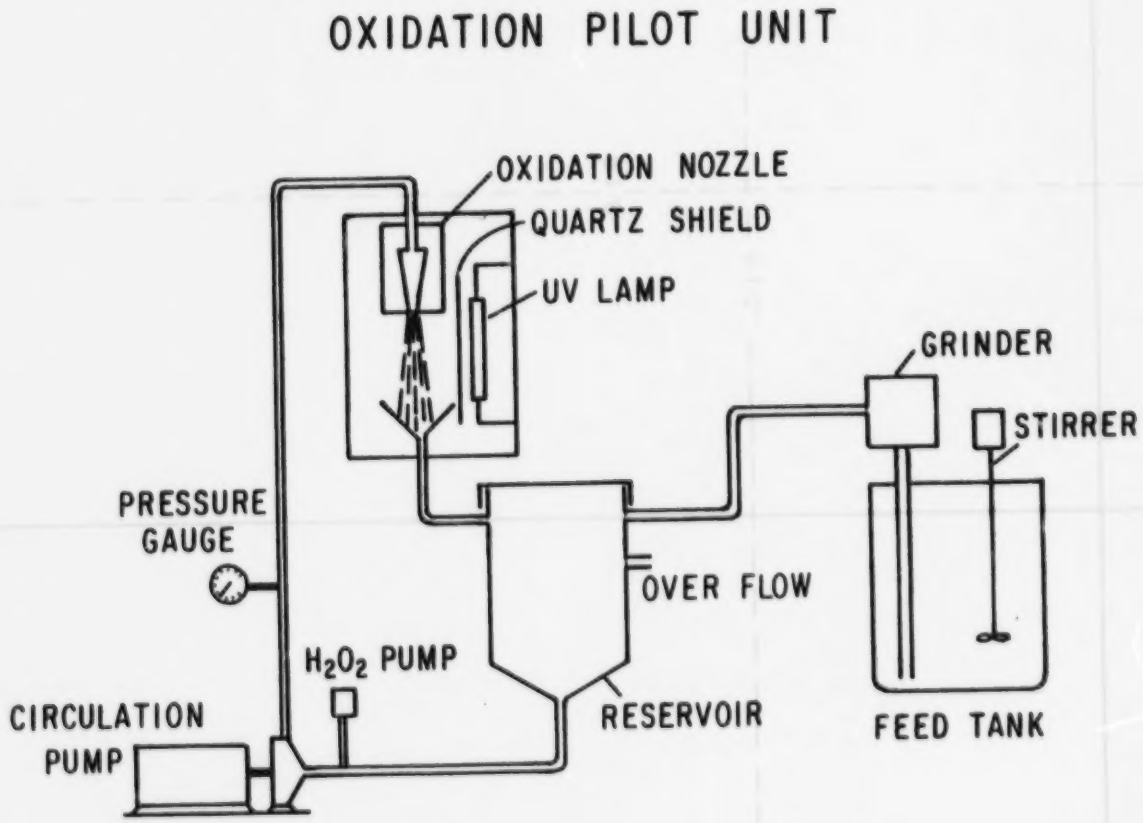

Figure 1. Schematic of Auto-Oxidation Pilot Plant.

SECTION 4

EXPERIMENTAL PROCEDURES

The original intention with this pilot plant was to run the effluent as it came from the beamhouse. The original material was to be pumped through a Moyno grinder pump into the reservoir and recycled through the oxidation loop back into the reservoir. The first serious problem encountered with this waste was excessive foaming. The high protein content of this waste is the probable cause of this problem. Within 1 min of cycling through the system without any pretreatment, the waste foamed over the top of the reservoir. After a large number of trials, Dow silicone antifoams A or B were found to be a partial solution to this problem. A 10- to 20-fold dilution of the incoming waste with water was necessary, in addition to adding the antifoam. A general observation made over the period of experimental trials was that as the length of time increased between production of the lime-sulfide unhairing waste in the tannery and the time it was finally tested in the oxidative system, its foaming characteristics increased and additional antifoam was needed to control it.

STATIC TESTS FOR REMOVALS

The removal of sulfide by this method was consistent and rapid in the static tests. Initial sulfide concentrations in these runs were between 150 to 500 ppm. In less than 100 min, the sulfide concentrations were reduced to less than 2 ppm in each of the trial runs. This was consistent even when the pH dropped as low as 8.5. It is presumed that the sulfide was oxidized to sulfate. The changes in TKN, TOC, COD, suspended solids and volatile solids were small and not consistent. A small decrease was experienced in several runs, but the magnitude of this change was generally less than 15%. The results of two typical runs, at 15 to 1 and 20 to 1 dilution with water, are shown on Figures 2 and 3. Chemical oxygen demand was not determined in the static trials. In later tests TOC and COD invariably followed each other very closely. A static trial using sodium sulfide was run to determine the effect of a typical high concentration of sulfide, typical of a fresh unhairing solution. Figure 4 shows that in a static process, even in a full-strength sulfide solution, the sulfide is rapidly removed and presumably oxidized.

CONTINUOUS TEST FOR REMOVAL

Continuous waste treatment was done with a 10:1 dilution of the original waste. A continuous trial can be started in two ways. The reservoir and oxidation loop can be filled with the diluted waste solution to be treated or with tap water. It takes approximately 150 min at 0.5 gal/min to approach steady state between the solution being added and the

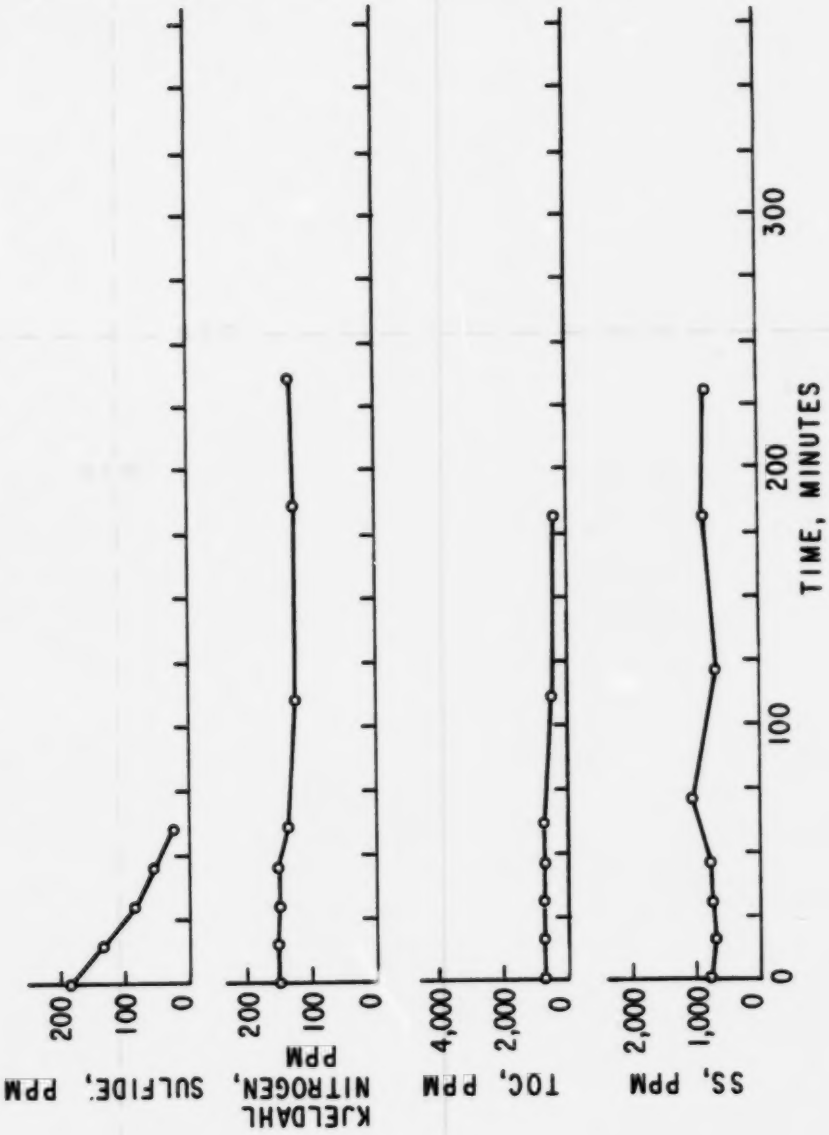

Figure 2. Results of Static Test in Pilot Plant. 15:1 Dilution of Unhairing Effluent.

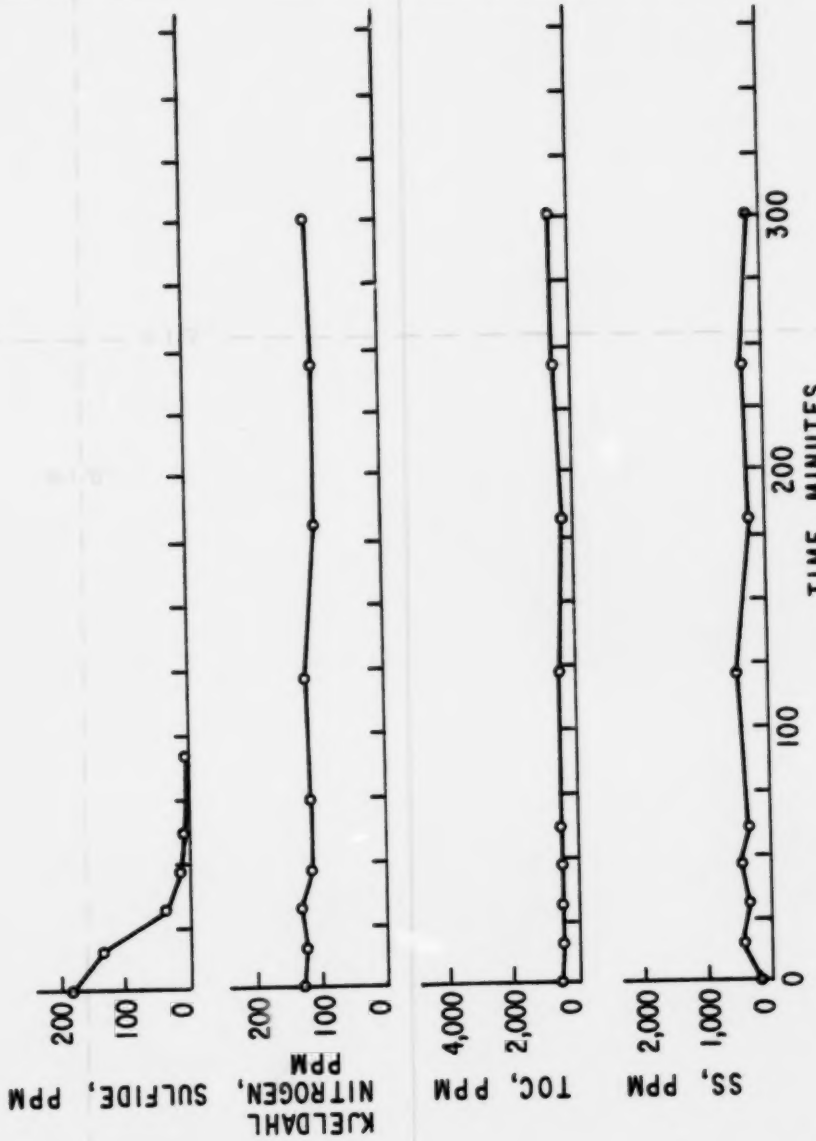

Figure 3. Results of Static Test in Pilot Plant. 20:1 Dilution of Unhairing Effluent.

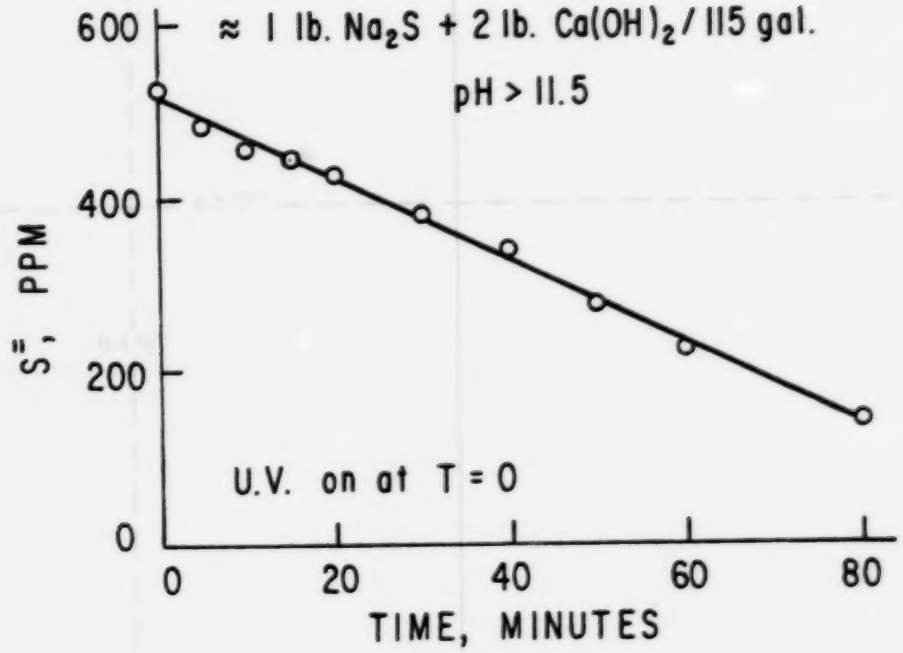

Figure 4. Reduction of Sulfide Concentration in Static Test of Pilot Plant on Sodium Sulfide/Lime Solution.

contents of the reservoir when the system starts with water. The results of two trials based on a 10:1 dilution are shown in Figures 5 and 6. Samples from the storage or feed tanks were also analyzed in the second run (Fig. 6). Sulfide reduction under these conditions was between 25 and 50%. Increasing the recycle ratio would probably result in further reduction. Again, little change in TOC, suspended solids, and volatile solids was observed, although there was a small overall reduction.

CONTINUOUS RUN WITH SETTLED WASTE

Continuous runs were repeated using a more conventional 2-hr settled waste as the starting material. The dilution was again 10:1. In these runs, samples were taken from the storage or feed tank as well as from the reservoir overflow. In Figure 7, only slight reductions were observed in any of the parameters when compared to those from the feed-tank solution. Within the scatter of the values obtained, a trend is not apparent.

ADDITION OF ULTRAVIOLET LIGHT AND/OR HYDROGEN PEROXIDE

Tannery unhairing effluent is a particularly difficult waste to handle even by conventional means due to its high pH, alkalinity, and relatively high protein content. Since the oxidative process alone was not effective in reducing COD, it was decided that the effect of additional energy input in the form of ultraviolet light and/or hydrogen peroxide might have a synergistic effect on the breakdown of this material and removal of COD. A preliminary bench-top experiment was performed to examine the effect of addition of peroxide, ultraviolet light, and copper ions to tannery lime-sulfide unhairing solutions. The procedure followed was to place 1 liter of unhairing waste in a beaker with constant stirring under a Phipps-Bird apparatus. Samples were then taken at 0, 15, 36, 90, and 120 min and at 20 hr. Samples were analyzed for pH, sulfide, total Kjeldahl nitrogen, TOC, COD, suspended and volatile solids. The control samples showed no change (Fig. 8). Addition of ultraviolet light to the solution did not alter values over the 20 hr period (Fig. 9). About half of the sulfide disappeared overnight with an apparent slight loss of nitrogen. Addition of 200 ml of 6% hydrogen peroxide dropped the sulfide concentration to 0 within 15 min. All other changes were negligible (Fig. 10). Combining ultraviolet light and hydrogen peroxide produced the same result (Fig. 11). Addition of copper sulfate to the raw waste did not change the values from that found without any additions (Fig. 12). That is, the sulfide dropped this time about 80% overnight, the other values remaining constant. Raw waste, ultraviolet light, and copper resulted in the same analyses (Fig. 13). Addition of peroxide to the copper again resulted in sulfide removal within 15 min. After an apparent loss of nitrogen and TOC at 15 min, the solution stabilized (Fig. 14). No appreciable change was observed in the experiment where ultraviolet light was added to the copper and peroxide (Fig. 15). The net result of these experiments demonstrates only that peroxide additions reduced sulfide very rapidly. Effects from copper and ultraviolet light additions were not observed. It should be mentioned here that concurrent work in our laboratory (Chang, unpublished), using intense ultraviolet radiation in intimate contact with the same type of waste, caused significant decreases in COD in the same time periods.

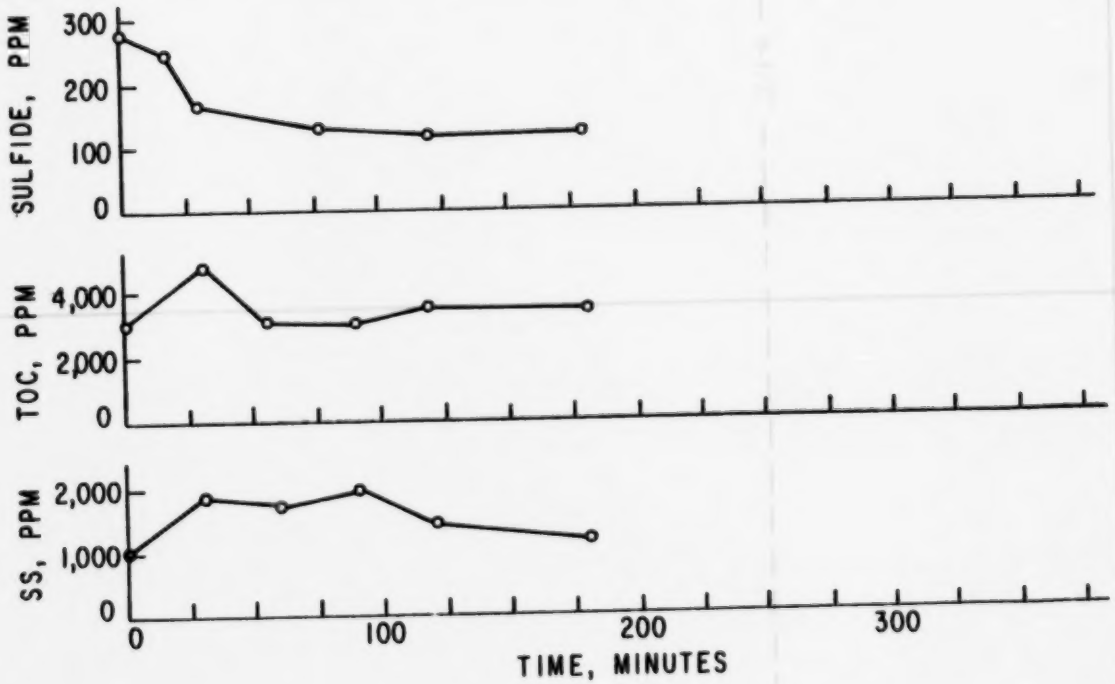

Figure 5. Results of Continuous Feed Test in Pilot Plant. 10:1 Dilution of Unhairing Effluent

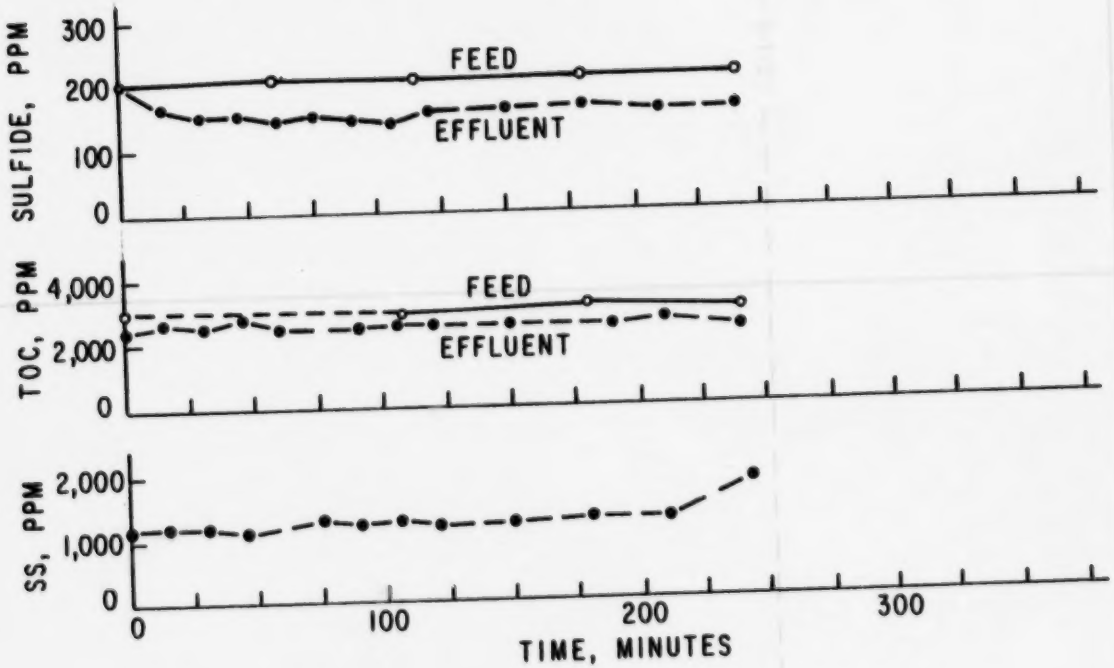

Figure 6. Results of Continuous Feed Test in Pilot Plant. 10:1 Dilution of Unhairing Effluent. Analysis of Feed for Sulfide and TOC.

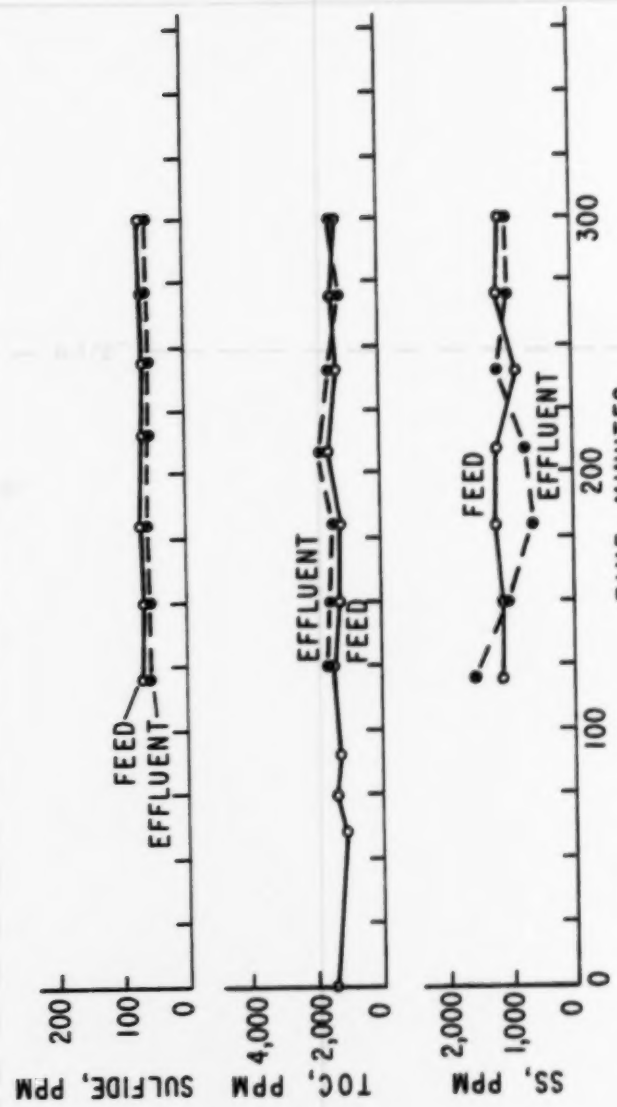

Figure 7. Results of Continuous Feed of Settled Unhairing Effluent. Diluted 10:1.

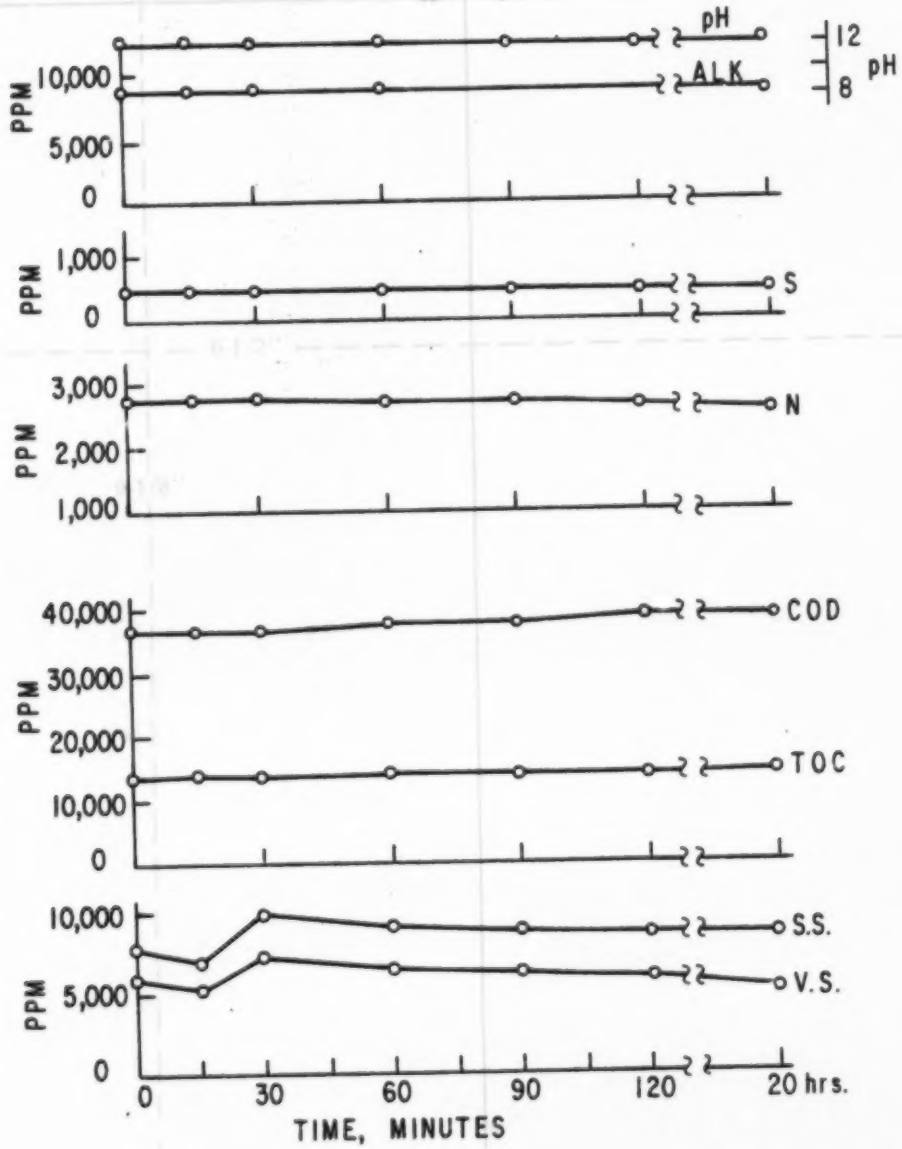

Figure 8. Bench Test of Additives to Tannery Waste. Control.

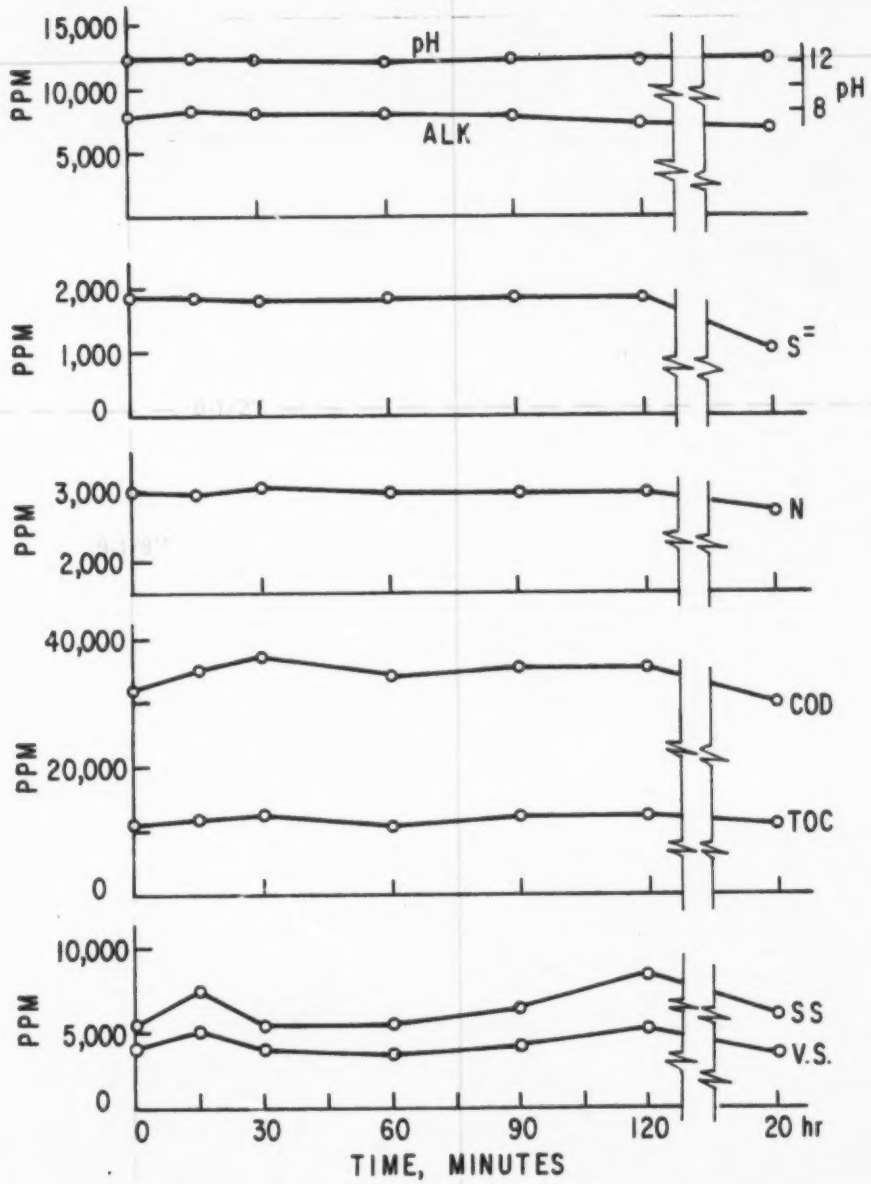

Figure 9. Bench Test of Additives to Tannery Waste Effect of Ultraviolet Light.

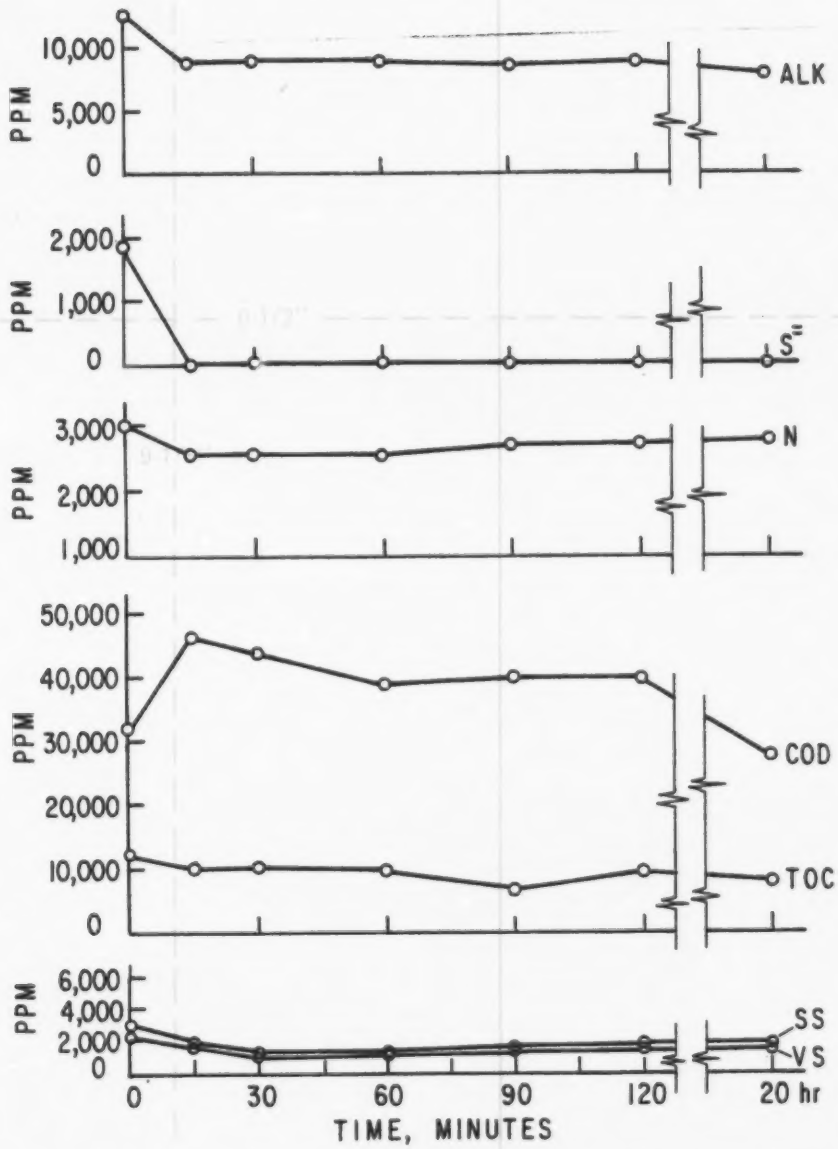

Figure 10. Bench Test of Additives to Tannery Waste. Effect of Hydrogen Peroxide.

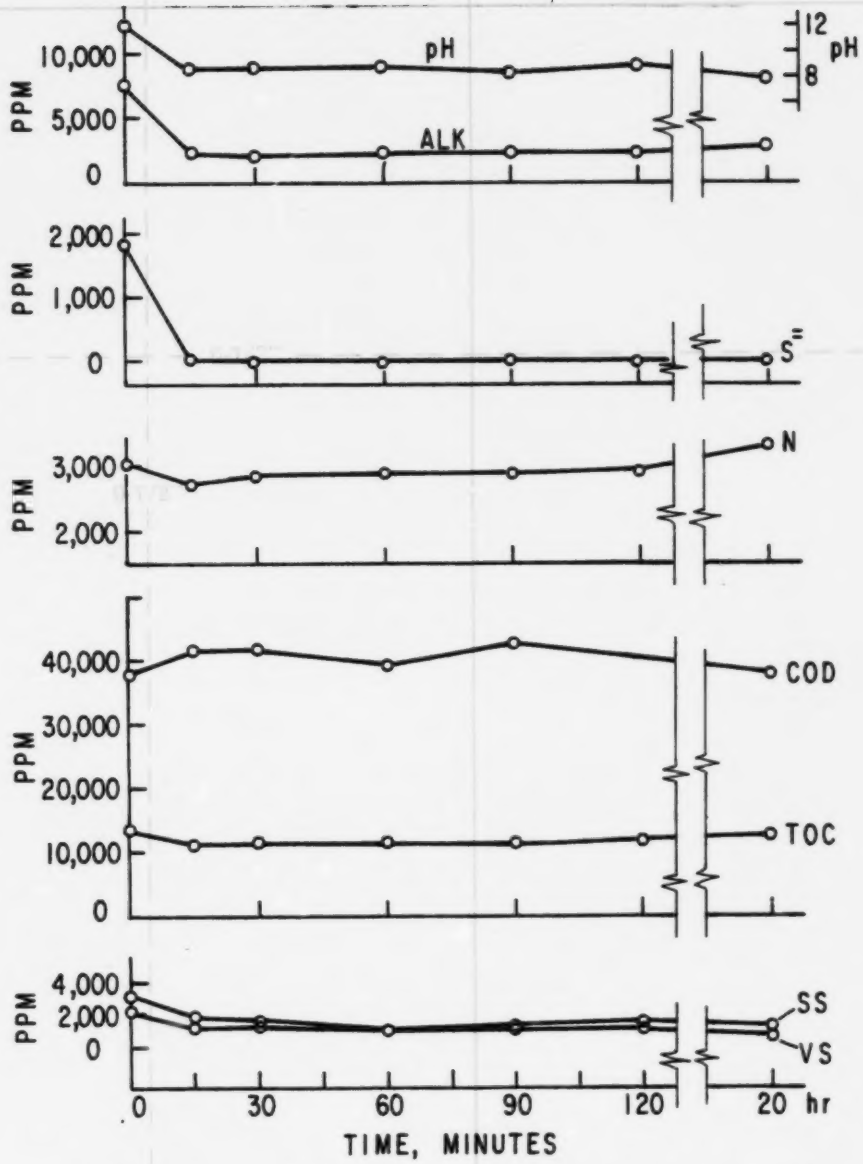

Figure 11. Bench Test of Additives to Tannery Waste Effect of Ultraviolet Light and Hydrogen Peroxide.

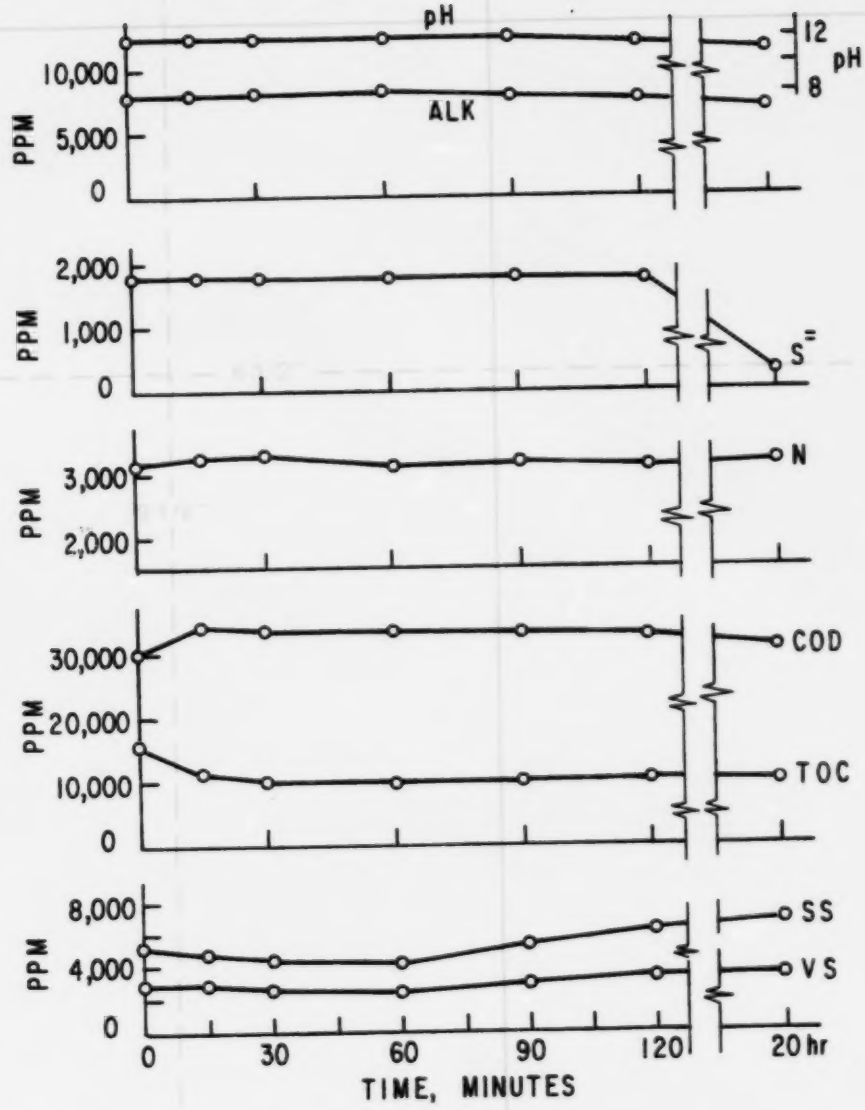

Figure 12. Bench Test of Additives to Tannery Waste. Effect of Copper Catalyst.

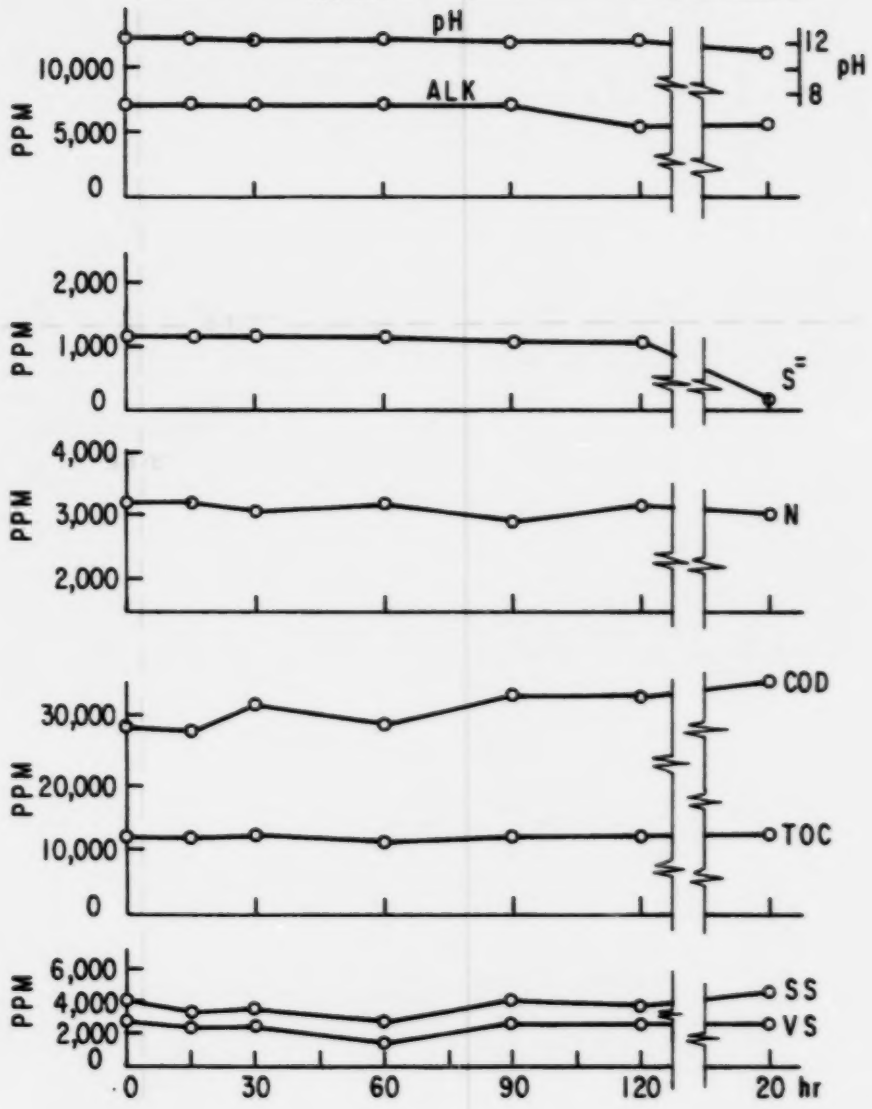

Figure 13. Bench Test of Additives to Tannery Waste. Effect of Ultraviolet Light and Copper Catalyst.

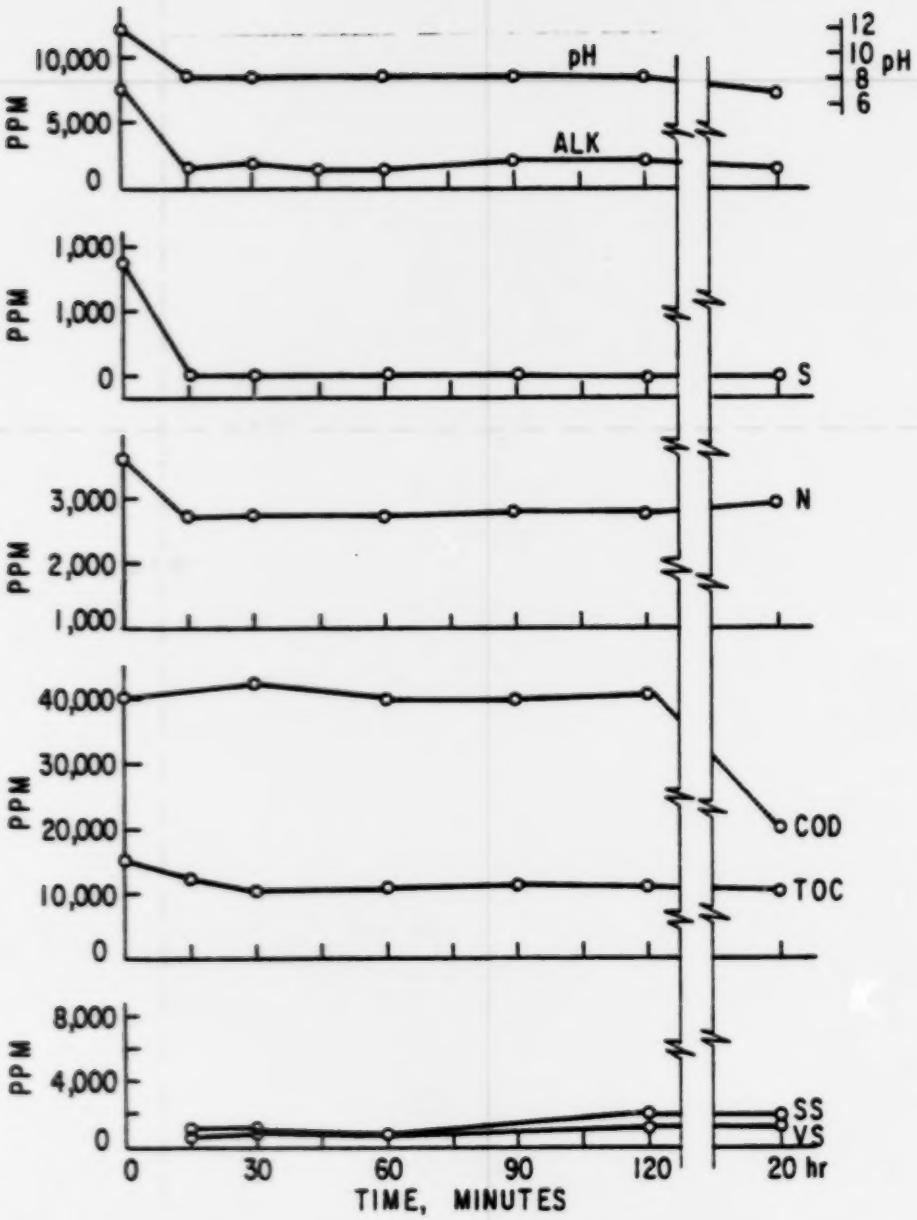

Figure 14. Bench Test of Additives to Tannery Waste. Effect of Hydrogen Peroxide and Copper Catalyst.

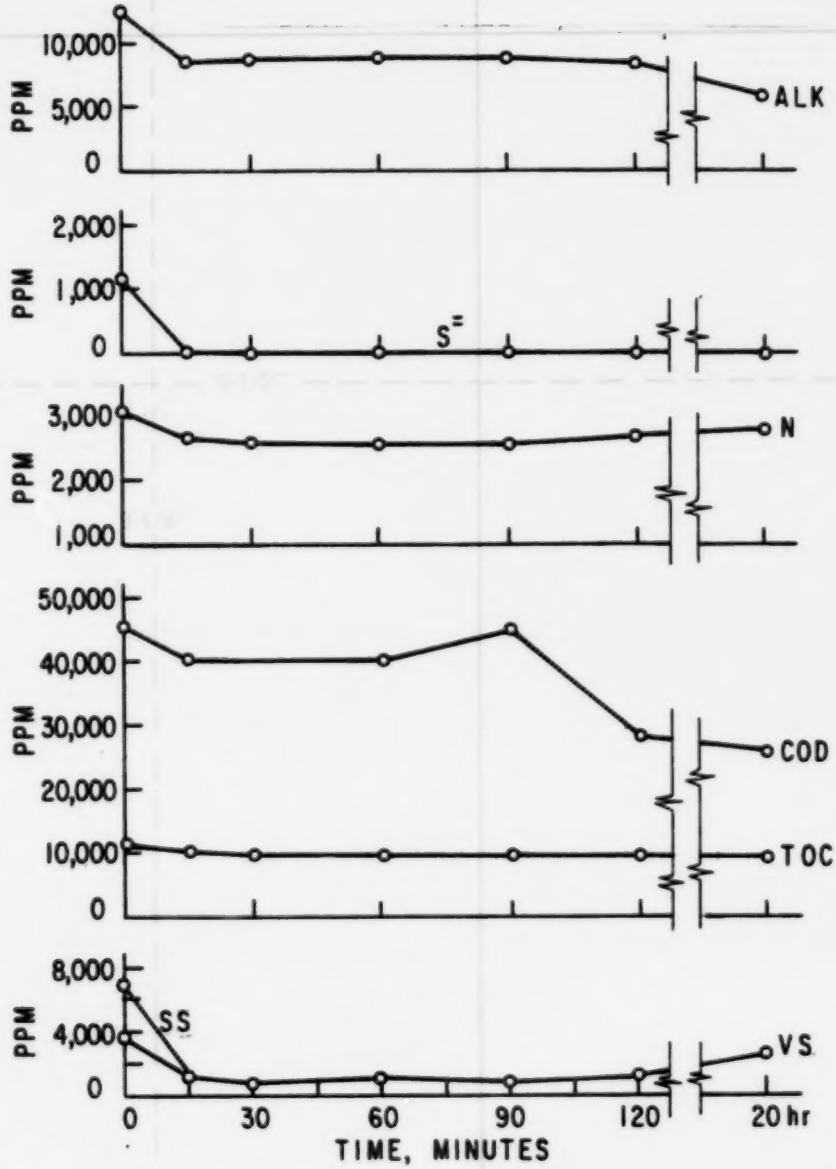

Figure 15. Bench Test of Additives to Tannery Waste. Effect of Hydrogen Peroxide, Ultraviolet Light and Copper Catalyst.

ADDITION OF ULTRAVIOLET AND HYDROGEN PEROXIDE TO THE OXIDATIVE PROCESS

The ultraviolet lamp, as shown in Figure 1, was added to the system toward the end of the project. Its location was designed to effect the solution immediately as it left the oxidation nozzle. The addition of the ultraviolet lamp was expected to assist with the breakdown of the materials being treated. A quartz shield was installed to keep water from splashing on the lamp. Figures 16 and 17 reflect the change in COD occurring in the effluent from a continuous run using ultraviolet light and hydrogen peroxide. In both trials, the ultraviolet lamp was turned on at the beginning of the run and peroxide added at the times indicated. In each case, the addition of peroxide reduced the COD by approximately 50 ppm. The last experiments run before the termination of this agreement were similar to the previous two, except for the additions of ultraviolet light and hydrogen peroxide. In these experiments, COD, TOC, and nitrogen were followed. In the experiment in Figure 18, the ultraviolet light was turned on 3 hr after the run was started. The reservoir in these experiments was filled with water and then at pH 11, 1:20 dilution of beamhouse effluent was added continuously. The nitrogen data demonstrate that the system is in steady state at 180 min. No change occurred in the nitrogen levels in either experiment. In the first experiment, peroxide was added after the ultraviolet light had been on for 3 hr. In the second experiment (Figure 19), peroxide was added at 180 min and the ultraviolet lamp was added 3 hr after the peroxide addition. The scatter for the values for TOC and COD was quite large. Considering the remarkably uniform analysis for nitrogen in the same samples, this result is unexpected. However, in spite of this scatter, the data suggest that reduction of COD and TOC occurred after addition of peroxide and that the u.v. light had little or no effect regardless of the order of addition. The data show that reduction of COD is approximately 25% when peroxide is added over a 6-hr period and 12% when added for half that time or for only 3 hr. This does not suggest a role for ultraviolet light in this particular demonstration. Again, the same point can be made here that was made in connection with the beaker test. The previously cited experiments by Chang demonstrated that ultraviolet light can break down this type of waste; however, the penetration of the ultraviolet light under these particular test conditions was insufficient to demonstrate that same effect.

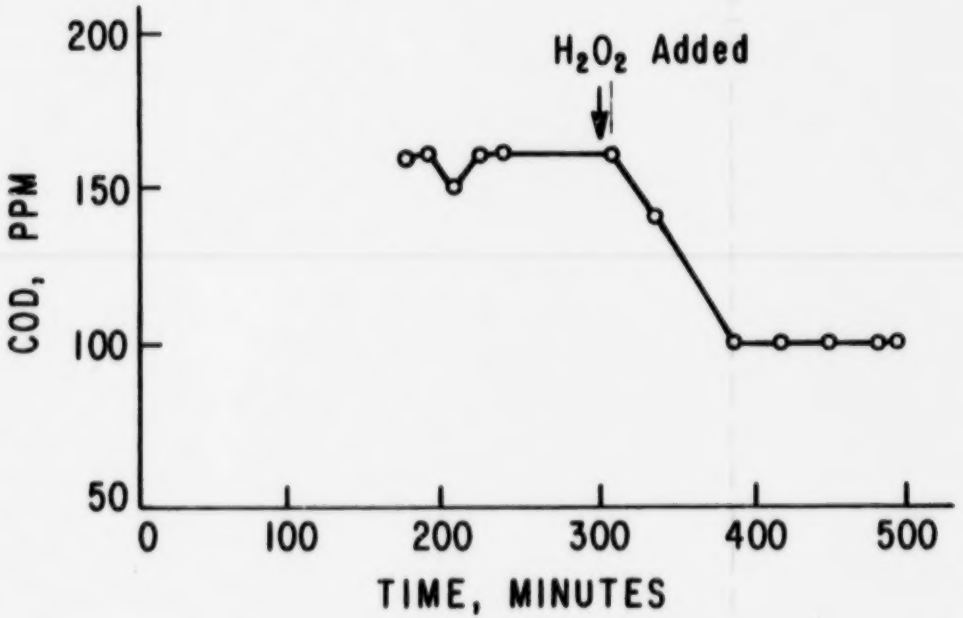

Figure 16. Effect of Addition of Hydrogen Peroxide to a Continuous Feed Pilot Plant Test. 10:1 Dilution of Unhairing Effluent.

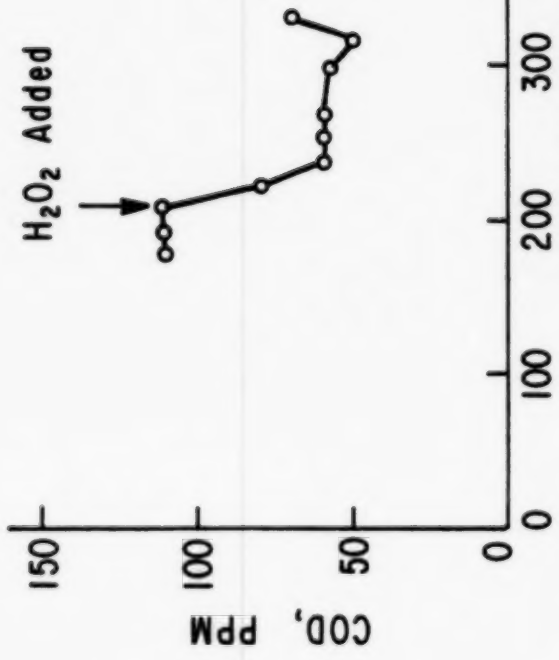

Figure 17. Effect of Addition of Hydrogen Peroxide to a Continuous Feed Pilot Plant Test. 20:1 Dilution of Unhairing Effluent.

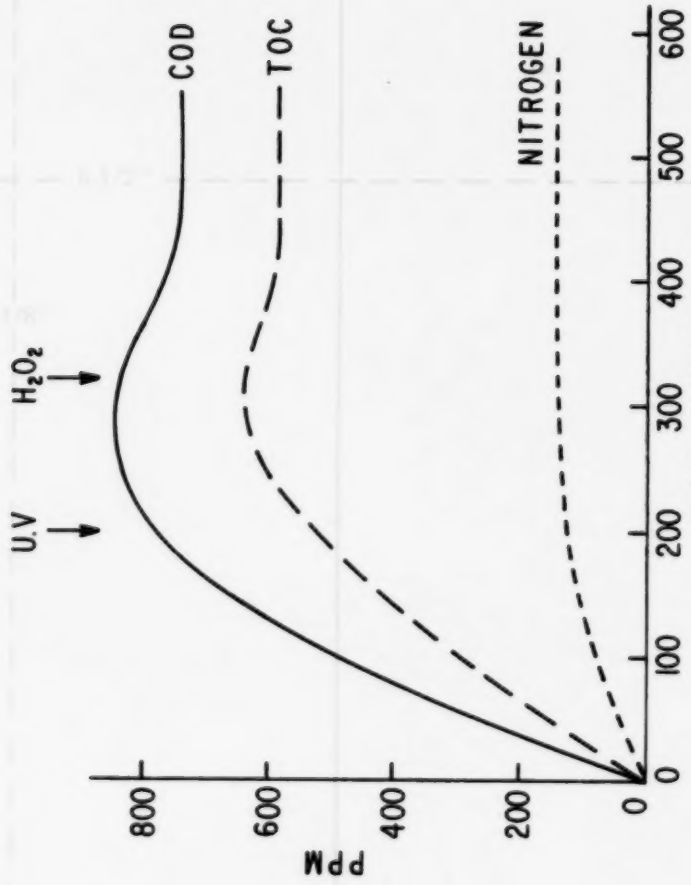

Figure 18. Effect of Addition of Ultraviolet Light Followed by Hydrogen Peroxide to a Continuous Feed Pilot Plant Test. 10:1 Dilution of Unhairing Effluent.

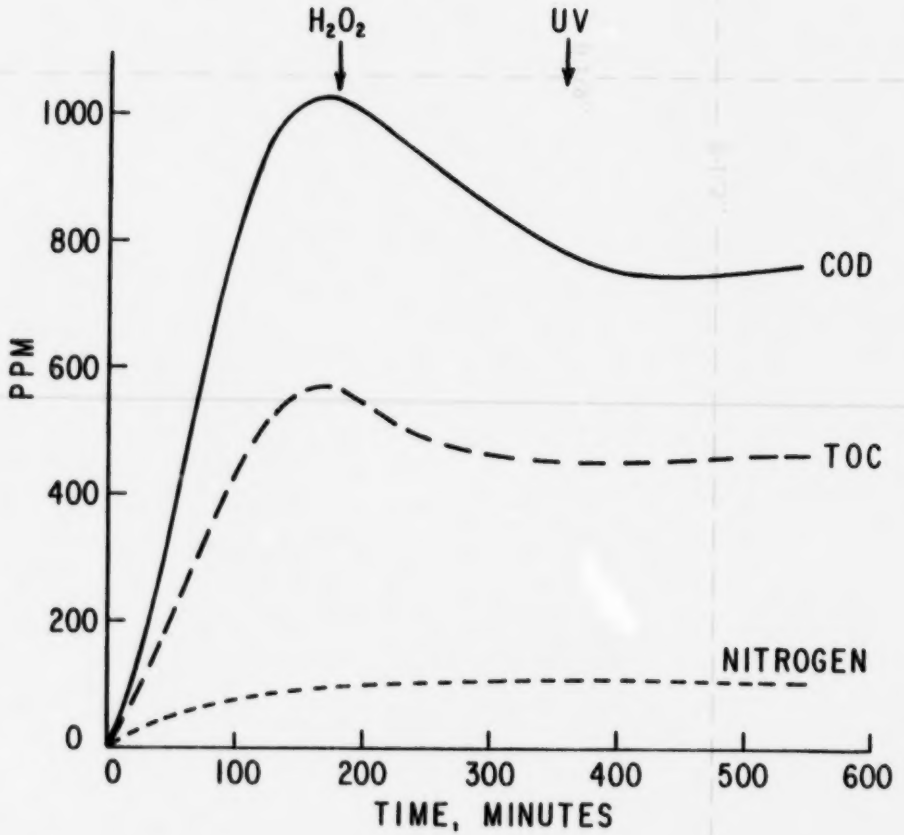

Figure 19. Effect of Addition of Hydrogen Peroxide Followed by Ultraviolet Light to a Continuous Feed Pilot Plant Test. 10:1 Dilution of Unhairing Effluent.

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